

# PATENT SPECIFICATION

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## (54) PROCESS FOR THE PREPARATION OF LUBRICATING OILS

(71) We, SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V., a company organised under the laws of The Netherlands, of 30 Carel van Bylandtlaan, The Hague, The Netherlands, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for the preparation of lubricating oil with a high viscosity index.

Lubricating oils with a high viscosity index can be prepared by catalytic hydrocracking of a high-boiling mineral oil fraction, for example a vacuum distillate boiling at between 350 and 550°C or a deasphalted residual oil. After hydrocracking the hydrocarbons boiling below the range between 350 and 400°C are removed by distillation of the resultant product and the higher-boiling residue is dewaxed, for example by treatment with a mixture of methyl ethyl ketone and toluene at low temperature. The dewaxed residue constitutes a lubricating oil possessing good qualities, including a high viscosity index, for example between 100 and 140.

However, such processes suffer from the disadvantage that any quantity of waxes produced by the separation of the residue from the distillation of the effluent from the hydrocracking stage represents a loss of useful products and thus reduces the yield from the desired conversion.

Applicants have found that it is possible to improve the overall yield from such a conversion by subjecting the waxes obtained by separation of the hydrocracking product to catalytic hydroisomerization, utilizing a catalyst containing one or more metals of the iron group, one or more metals of Group VIB of the Periodic Table of Elements, boron and a catalyst carrier consisting of oxide.

Moreover, Applicants have found that the said catalytic hydro-isomerization process represents not only an important means of

improving the overall yield in the production of lubricating oils from a high-boiling mineral oil fraction, but also a very attractive means of producing lubricating oils having a viscosity index even higher than that of lubricating oil produced by hydrocracking, for example between 140 and 170.

In order to derive benefit from this latter advantage, it is necessary to precisely select the feedstock to the hydro-isomerization reactor, as will be described below in greater detail.

According to the present invention a process for the preparation of lubricating oil with a high viscosity index is provided, wherein a high-boiling mineral oil fraction is subjected to catalytic hydrocracking treatment at elevated temperature and pressure and in the presence of a hydrocracking catalyst and hydrogen, the hydrocarbons boiling below a temperature between 350 and 400°C are separated from the liquid product of the hydrocracking treatment, the residue boiling above a temperature between 350 and 400°C is separated by dewaxing into lubricating oil with a high viscosity index and wax, said wax is subjected to catalytic hydro-isomerization in the presence of hydrogen and a catalyst containing one or more metals of the iron group, one or more metals of Group VIB of the Periodic Table of Elements, boron and a catalyst carrier consisting of oxide, and lubricating oil with a high viscosity index is separated from the catalytic hydro-isomerization product by dewaxing at least that part of said product which consists of hydrocarbons boiling within the boiling range of lubricating oils.

Starting materials suitable for the process according to the invention are mixtures of high-boiling hydrocarbons, for example heavy petroleum fractions and heavy fractions produced by pyrolysis of coal, bituminous shale or tar sand. Petroleum fractions boiling, at least in part, above the boiling range of lubricating oil can be used to advantage. As feedstock for the present pro-

cess, preference is given to a fraction obtained by vacuum distillation of a petroleum residue obtained by atmospheric distillation. The boiling range of such a vacuum distillate is generally between 350 and 550°C. However, deasphalted residual petroleum fractions are particularly preferred. Mixtures of vacuum distillates and deasphalted residual petroleum fractions are equally suitable for use.

The hydrocracking treatment and the hydro-isomerization according to the invention are carried out at elevated temperature and pressure in the presence of hydrogen or a hydrogen-containing gas. Pure hydrogen could be used, but this is unnecessary. A gas having a hydrogen content of 70% or more is perfectly suitable.

In practice, use will preferably be made of a hydrogen-containing gas originating from a catalytic reforming plant. Such a gas not only has a high hydrogen content but also contains low-boiling hydrocarbons, such as methane, ethane and a small amount of propane.

The temperature and pressure used in the hydrocracking treatment may vary between very wide limits, depending on the desired degree of conversion. A temperature will generally be selected not lower than 300°C and not higher than 550°C. At temperatures lower than 300°C the rate of conversion is reduced, whereas at temperatures higher than 550°C forced cracking takes place, such that only a limited quantity of the desired product is obtained. A temperature between 350 and 450°C is to be preferred, a temperature between 350 and 400°C being particularly preferred. Pressures lower than 50 bar are less desirable since they shorten the life of the catalyst and bring with them the risk of an excessive level of aromatics in the product, which would adversely affect both the viscosity index and the final properties of the product. A pressure exceeding 250 bar would require very costly plant. Preference is therefore given to an absolute pressure between 100 and 200 bar.

As regards the space velocity and hydrogen/oil ratio, these may also be selected within very wide limits. However, a space velocity between 0.1 and 5 kg of oil per hour per litre of catalyst is preferably selected. A space velocity of less than 0.1 kg/h.l would require such a large reactor to convert a given quantity that it would be uneconomic, whereas a space velocity greater than 5 kg/h.l would only achieve a low rate of conversion to the desired product.

The hydrogen/oil ratio is preferably between 100 and 5000 standard litres (litres at 1 bar and 0°C) per kg of oil. A very low H<sub>2</sub>/oil ratio would adversely affect the life of the catalyst, whereas a very high H<sub>2</sub>/oil ratio would cause a considerable loss of feedstock over the catalyst beds, requir-

ing high compression to circulate the gas with high hydrogen content.

The hydrocracking catalysts used in the process according to the invention are catalysts containing one or more hydrogenating constituents on a carrier. The hydrocracking catalysts preferably consist of at least one or more metals of Groups VB, VIB, VIIB and/or VIII of the Periodic Table of Elements and/or one or more oxides and/or sulphides of one or more of these metals. Preferably the only such metals present (as such and/or as oxides and/or sulphides) are nickel and tungsten.

Groups VB, VIB, VIIB and VIII of the Periodic Table of Elements comprise the following metals, respectively: V, Nb, Ta, Cr, Mo, W, Mn, Tc, Re, Fe, Co, Ni, Ru, Rh, Pd, Os, Ir and Pt. Suitable hydrocracking catalysts are those containing at least two metallic hydrogenating components, one of these metallic components preferably consisting of nickel and/or cobalt and/or a compound of nickel and/or cobalt, the other consisting of molybdenum and/or tungsten and/or a compound of molybdenum and/or tungsten. Important factors in this process are the amount of metallic hydrogenating component, expressed as a percentage of the total catalyst, and the ratio between the nickel and/or cobalt on the one hand and the molybdenum and/or tungsten on the other.

It is advantageous not to select too small a quantity of metallic component, since this could affect the required hydrogenating action. A very large amount of metallic component would result in the pores of the porous catalyst carrier becoming clogged, such that part of the specific surface area of the catalyst would be rendered inaccessible to the material to be converted. It has been found that a catalyst containing 3—25% by weight of molybdenum and/or tungsten lends itself admirably to the process according to the invention. Preference is therefore given to applying the above-mentioned weight percentages of metallic hydrogenating components in the catalyst before the latter is used for the present process.

The activity and selectivity of these latter catalysts can be further improved by adding phosphorus, for example between 1 and 10% by weight of phosphorus calculated as P<sub>2</sub>O<sub>5</sub>. A phosphorus content between 2 and 9% by weight, calculated as P<sub>2</sub>O<sub>5</sub>, based on the total catalyst, is even more preferred, and it has been found that maximum catalyst activity is obtained when the phosphorus content is between 3 and 7% by weight, likewise calculated as P<sub>2</sub>O<sub>5</sub>, based on the total catalyst. A preferred hydrocracking catalyst contains 3—25% wt. of nickel and/or cobalt, 6—24% wt. of molybdenum and/or tungsten and 2—9% wt. of phosphorus, calculated as P<sub>2</sub>O<sub>5</sub>.

Another preferred group of hydrocracking catalysts is represented by the group of hydro-isomerization catalysts described below. In this case the most suitable hydrocracking catalyst is a catalyst which is completely identical to the hydro-isomerization catalyst, thus representing a considerable simplification.

Generally speaking, the hydro-isomerization catalyst used in the process according to the invention contains at least two metallic hydrogenating components, a carrier and boron. One of the metallic components consists of at least one metal of the iron group (Fe, Co, Ni) or a compound of such a metal; moreover, the catalyst must contain at least one metal of Group VIB or a compound of such a metal.

Although all the metals of the iron group and all the metals of Group VIB of the Periodic Table of Elements may be used as one of the two hydrogenating components in the catalyst for the conversion according to the invention, a combination of nickel and tungsten, which makes it possible to achieve the best results, is generally preferred. As regards the weight percentage of the metallic constituents, preference is given to hydro-isomerization catalysts containing 3—16% by weight of one or more metals of the iron group and 6—24% by weight of one or more metals of Group VIB.

The amount of boron used on the catalyst may also vary between wide limits. However, Applicants have found that the activity of the catalyst depends on the percentage of weight of boron contained in the latter. A boron content of between 1 and 20% by weight, calculated as  $B_2O_3$ , provides good catalyst activity. A catalyst having such a boron content is therefore used. Even greater preference is given to a boron content between 3 and 15% by weight, calculated as  $B_2O_3$ , based on the total catalyst, and it has been found that maximum catalyst activity is obtained when the boron content is between 5 and 10% by weight, likewise calculated as  $B_2O_3$ , based on the total catalyst.

As hydrocracking and/or hydro-isomerization catalyst carrier, use can in principle be made of any refractory material which is resistant to the chemical products. Suitable materials are, for example, alumina, silica, magnesia, titania and mixtures and compounds of these oxides.

Commercial alumina generally contains small amounts of impurities, for example silica and sodium. It has been found that alumina containing between 0.1 and 3%, preferably 0.5 and 3%, by weight of silica and a maximum of 0.01% and preferably 0.005% by weight of sodium is an excellent carrier material for the catalyst. Preference is therefore given to this alumina. If the alumina contains more than 0.005% by weight of sodium, this

sodium content can be reduced to the required level by ion exchange with a solution of ammonium salts. Finally, use can also be made of acidic carriers, such as acid-treated clays and zeolitic molecular sieves.

To further increase the activity of the hydro-cracking and/or hydro-isomerization catalysts, from 1 to 6% by weight (based on the carrier) of fluorine is preferably added. However, it should be noted that, if the hydrocracking catalyst employed (whether or not fluorinated) contains nickel and/or cobalt, molybdenum and/or tungsten and phosphorus, the use of hydro-isomerization catalyst containing fluorine, is not envisaged.

The hydrocracking and hydro-isomerization catalysts can be prepared in any desired manner, for example by impregnating the carrier with one or more aqueous solutions of compounds of the other components, drying it and calcining it in the ranges of temperature, respectively, of 100—250°C and 450—850°C, for a period of 0.5—5 hours.

After calcination the metallic constituents and, if used, the phosphorus will probably be present in the form of oxides in the catalyst, although the possibility cannot be excluded of their being at least partially bound to the catalyst carrier.

It has been found that the catalytic conversion of heavy hydrocarbon mixtures to lubricating oil by means of hydrogen gives better results with previously sulphided hydro-cracking and/or hydro-isomerization catalysts, the metallic oxides of which have been converted, at least in part, to corresponding metallic sulphides.

Preference is therefore given to a sulphided catalyst. Although the feedstock to the hydro-cracking zone generally contains sulphur compounds which rapidly sulphide an oxide-containing catalyst after the start of the hydro-cracking treatment, it is advantageous to previously sulphide the hydrocracking catalyst so that a sulphided catalyst is utilized from the start of the hydrocracking process.

A particularly good method, and therefore one preferably used for sulphiding, consists in contacting the catalyst at a temperature between 250°C and 450°C and a pressure between 30 and 70 bar—the space velocity being between 1 and 10 kg of oil per hour per litre of catalyst and the hydrogen/oil ratio being between 50 and 500 standard litres of  $H_2$  per kg of oil—with an oil containing sulphur compounds, and preferably with gas oil containing sulphur compounds. This treatment is preferably carried out in the same reaction vessel in which the hydro-generative conversion to lubricating oil is carried out.

After sulphiding of the catalyst, the starting material for the production of lubricating oil with a high viscosity index is introduced into the reactor at a suitable tempera-

ture, pressure, space velocity and  $H_2$ /oil ratio, and is passed over the catalyst which is preferably contained in one or more beds consisting of particles with dimensions

5 between 0.5 and 5 mm.  
After passing through the hydrocracking reactor, the product is cooled and separated into a gas with a high hydrogen content and a liquid product. The gas with a high  
10 hydrogen content is preferably recycled, at least in part, to the reactor. The liquid product contains hydrocarbons boiling below the boiling range of lubricating oil and hydrocarbons boiling within the boiling range of  
15 lubricating oil.

The hydrocarbons boiling below the boiling range of lubricating oil are preferably separated by fractional distillation of the higher boiling residue. The cut point of this  
20 distillation is selected such that the initial boiling point of the higher boiling residue is between 350 and 400°C.

Apart from excellent lubricating oil components, this residue contains normal and slightly branched paraffins designated by the term "wax", which solidify at ambient temperature and thus have an adverse effect on the pour point of the desired lubricating oil. This residue is therefore dewaxed to form  
30 a lubricating oil or a usable lubricating oil component. This treatment can be carried out in any desired manner, for example by means of a solvent. This latter process consists in dissolving a feedstock containing this  
35 wax in an organic solvent and chilling this

feedstock to cause crystallization of the wax, which is subsequently separated from the solvent/oil mixture by filtration. Suitable solvents for this treatment are liquid propane, butane, pentane, benzene, toluene, acetone,  
40 methyl ethyl ketone and mixtures of one or more aromatics with methyl ethyl ketone.

Dewaxing is preferably carried out by means of a mixture of 40—60 parts by volume of methyl ethyl ketone and 60—40  
45 parts by volume of toluene at a temperature between -10 and -30°C, the volume ratio of the solvent and oil being between 1:1 and 10:1.

The dewaxed residue possesses a high viscosity index, for example between 100 and 140, depending on the conditions in which the hydrogenating conversion of the starting material has been carried out. It is thus eminently suitable for use as a  
55 "multigrade" lubricating oil or as a component of "multigrade" lubricating oil. This dewaxed residue may moreover serve for the preparation by vacuum distillation of one or more lubricating oils or lubricating oil components with high viscosity indices and varying viscosities, which may subsequently be converted into excellent "multigrade"  
60 lubricating oils by mixing them together or with other components.

The wax obtained from the dewaxing process is subsequently subjected to catalytic hydro-isomerization, preferably under the following reaction conditions:

70	Temperature:	310—450°C
	Absolute pressure:	50—200 bar, preferably 90—200 bar
	Space velocity:	0.1—5.0 l of wax per hour per litre of catalyst
75	$H_2$ /wax ratio:	100—5000 standard litres of $H_2$ (0°C, 1 bar) per kg of wax

Since the conversion in the hydro-isomerization zone is incomplete, it is necessary to separate from the unconverted wax, the oil obtained as a result of the conversion, which  
80 is effected by dewaxing.

Small amounts of hydrocarbons boiling below the boiling range of lubricating oils may be present in the effluent from the hydro-isomerization zone, which is dewaxed again in a second dewaxing zone; however,  
85 it is preferable to exclude these materials since they act as an undesirable diluent and thus reduce the efficiency and yield of the later treatment stages. Consequently, the feedstock for the second dewaxing zone preferably consists only of the fraction boiling within the boiling range of lubricating oils in the effluent from the isomerization zone. All of the normally liquid fraction, comprising neopentane and the higher-boiling hydrocarbons, of the effluent from the hydro-iso-

merization zone, or any selected part of this fraction, may nevertheless be used in the second dewaxing zone provided that the part boiling within the boiling range of lubricating  
100 oils is included.

The term "hydrocarbons boiling within the boiling range of lubricating oils" refers to high-boiling hydrocarbons having a viscosity which makes them useful as lubricants or lubricant components. Although some lubricating oils may be more volatile, most lubricating oils have an initial boiling point at atmospheric pressure of at least 350°C.

The separation of the hydrocarbons boiling below the boiling range of lubricating oils in the effluent from the hydro-isomerization zone is conveniently effected by fractional distillation and/or flash separation. However, other separation processes may also be used.  
115

The dewaxing of the fraction boiling within the boiling range of lubricating oils,

which has been obtained from the effluent from the isomerization zone, may be effected by conventional means. Solvent dewaxing may conveniently be used for this purpose.

- 5 In this operation a waxy feedstock is dissolved in an organic solvent and is subsequently chilled to cause crystallization of the wax which is separated by filtration from the solvent/oil mixture. Suitable solvents for this operation are liquid propane, benzene, toluene, acetone, methyl ethyl ketone and mixtures of methyl ethyl ketone with one or more aromatics.

- 10 The dewaxing of the fraction boiling within the boiling range of lubricating oils in the effluent from the hydro-isomerization zone is preferably effected by means of a mixture of 40—60 parts by volume of methyl ethyl ketone and 60—40 parts by volume of toluene at a temperature between  $-10$  and  $-40^{\circ}\text{C}$ , the volume ratio of the solvent and oil being between 1:1 and 10:1.

- 15 The waxes obtained after the dewaxing of the hydro-isomerization product are advantageously at least partially recycled to the hydro-isomerization zone or to the hydrocracking zone, this embodiment of the process according to the invention further improving the total yield from the intended conversion.

- 20 Furthermore, according to another embodiment of the process according to the invention, a mixture of the waxes obtained after hydrocracking and a part of the same fraction of high-boiling mineral oil to be converted by hydrocracking may be subjected to hydro-isomerization. In this way lubricating oils with a viscosity index between 100 and 140 are obtained after hydro-isomerization and dewaxing, while the total lubricating oil yield is between 35 and 80%, based on the total mineral oils feedstock, when all the waxes obtained after hydro-isomerization are recycled.

- 25 If the waxes obtained after hydrocracking are not mixed with a fraction of the mineral oils, but are converted as such by hydro-isomerization, lubricating oils with even higher viscosity indices, i.e. between 140 and 170, are obtained with the same extremely favourable conversion yields.

Lubricating oils with high or very high viscosity indices are eminently suited for use as "multigrade" lubricating oils or as components of "multigrade" lubricating oil. These products may serve for the preparation by vacuum distillation of one or more types of lubricating oil or lubricating oil component with very high viscosity indices and with varying viscosities.

The lubricating oil components may be converted into excellent "multigrade" lubricating oils by mixing them together or with other lubricating oil components.

In order to simplify the process according to the invention, it is recommended to carry out the hydrocracking treatment and the hydro-isomerization by means of the same catalyst. This catalyst may be divided between two separate reactors such that the hydrocracking treatment can be carried out in the first reactor and the hydroisomerization in the second.

However, it is possible to carry out the hydrocracking treatment and the hydro-isomerization in the same reactor. In this way only one reactor is required for the process according to the invention, which represents a considerable cost saving.

As already mentioned above, the wax which has been separated from the hydro-isomerization product may be recycled to the hydro-isomerization reactor or to the hydrocracking reactor for further conversion to lubricating oil with a very high viscosity index.

If the hydrocracking treatment and the hydro-isomerization are carried out in the same reactor, it is preferable to pass the wax separated from the hydro-isomerization product together with the high-boiling mineral oil fraction to the reactor and to subject the mixture of wax and high-boiling mineral oil fraction to a combined hydro-isomerization and hydrocracking treatment.

The invention may be illustrated more clearly by means of the following examples:

#### Example 1

A residual petroleum fraction, deasphalted by means of liquid propane, originating from a North African crude oil had the following properties:

105	Specific gravity 20/4: Viscosity at $37^{\circ}\text{C}$ : Viscosity index ( $\text{VI}_E$ ) ASTM-D 2270) after dewaxing at $-19^{\circ}\text{C}$ : Oil yield after dewaxing at $-19^{\circ}\text{C}$	0.907 32.8 cSt 77 89.2% by weight
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This deasphalted residual petroleum fraction was subjected to catalytic hydrocracking under the following reaction conditions:

- |    |                               |  |  |
|----|-------------------------------|--|--|
|    | Temperature:                  | 440°C  |  |
|    | Absolute pressure:            | 140 bar  |  |
| 5  | Space velocity:               | 1 kg of oil per hour per litre of catalyst           |  |
|    | H <sub>2</sub> /oil ratio     | 1000 standard litres of H <sub>2</sub> per kg of oil |  |
|    | Composition of catalyst used: | Al <sub>2</sub> O <sub>3</sub> 63.0% by weight       |  |
|    |                               | P <sub>2</sub> O <sub>5</sub> 3.9% by weight         |  |
| 10 |                               | MoO <sub>3</sub> 19.4% by weight                     |  |
|    |                               | NiO 9.7% by weight                                   |  |
|    |                               | F 3.0% by weight                                     |  |
|    |                               | SiO <sub>2</sub> 1.0% by weight                      |  |
- 15 56% by weight, consisting of the fraction boiling below 400°C, was separated by fractional distillation from the liquid product obtained by means of the hydrocracking treatment.
- 20 The fraction boiling above 400°C (44% by weight) was dewaxed by means of a mixture of 50 parts of methyl ethyl ketone and 50 parts of toluene at a temperature of -27°C. The solvent/oil ratio was 3:1. During this dewaxing treatment, 12% by weight of slack wax was separated. The lubricating oil thus obtained had a viscosity index (VI<sub>E</sub>, ASTM-D 2270) of 130.
- The yield of this lubricating oil was 38% by weight, based on the original deasphalted residual petroleum fraction.
- 30 The slack wax separated during dewaxing was subsequently subjected to catalytic hydro-isomerization under the following reaction conditions:
- |    |                            |  |  |
|----|----------------------------|--|--|
| 35 | Temperature:               | 340°C  |  |
|    | Absolute pressure:         | 140 bar  |  |
|    | Space velocity:            | 0.81 kg of slack wax per hour per litre of catalyst        |  |
|    | H <sub>2</sub> /oil ratio: | 1660 standard litres of H <sub>2</sub> per kg of slack wax |  |
- 40 The catalyst employed contained 67.5% by weight of Al<sub>2</sub>O<sub>3</sub>, 20.0% by weight of MoO<sub>3</sub>, 6.5% by weight of NiO and 6.0% by weight of B<sub>2</sub>O<sub>3</sub>. It had been prepared by impregnating alumina extrudates having a diameter of 1.5 mm with a solution of ammonia, ammonium para-molybdate, boric acid and nickel formate, subsequently drying the impregnated extrudates for 2 hours at 200°C and calcining them for 2 hours at 650°C.
- 45 This catalyst had previously been sulphided for 36 hours by contacting it, at a temperature of 350°C, a pressure of 50 bar, a space velocity of 1 kg per hour per litre of catalyst and a hydrogen/oil ratio of 150 standard litres of hydrogen per kg of oil, with heavy gas oil containing sulphur compounds.
- 50 This fraction boiling below 400°C was separated by fractional distillation from the isomerization product. The yield of residue boiling above 400°C was 45% by weight, based on the hydro-isomerization feedstock. This residue was dewaxed at -27°C by means of a mixture of methyl ethyl ketone and toluene (50/50), the solvent/oil ratio employed being 8:1.
- 55 The yield of dewaxed lubricating oil was 35% by weight, based on the hydro-isomerization feedstock.
- 60 The lubricating oil possessed the following properties:
- |    |  |          |  |
|----|--|----------|--|
|    | Viscosity index (VI <sub>E</sub> , ASTM-D 2270): | 158      |  |
| 75 | Kinematic viscosity at 38°C:                     | 30.6 cSt |  |
|    | Kinematic viscosity at 99°C:                     | 5.97 cSt |  |
- 80 Comparative test
- 85 A deasphalted residual petroleum fraction of the same origin and composition as the starting material in Example 1 was dewaxed at -27°C by means of a mixture of methyl ethyl ketone and toluene (50/50), the solvent/oil ratio employed being 3:1. In this operation 14% by weight of slack wax was separated off.
- 90 This slack wax was hydro-isomerized under the same reaction conditions as in Example 1 and using the same sulphided hydro-isomerization catalyst as described in Example 1.
- 95 The fraction boiling above 400°C of the liquid hydro-isomerization product was dewaxed in the same manner as that described for the hydro-isomerization product in Example 1.
- 70 The lubricating oil yield was now 20% by

weight, in relation to the hydro-isomerization feedstock. The lubricating oil obtained had the following properties:

5	Viscosity index (VI <sub>E</sub> , ASTM-D 2270):	100
	Kinematic viscosity at 38°C:	195 cSt
	Kinematic viscosity at 99°C:	16.89 cSt

#### Example 2

A mixture of 40% by weight of the de-asphalted residual petroleum fraction described in Example 1 and 60% of the slack waxes obtained by dewaxing the residue from the separation by distillation of the hydro-cracking product, as described in Example 1, was hydro-isomerized by means of the catalyst containing nickel, molybdenum and boron described in Example 1, under the following reaction conditions:

20	Temperature:	424°C
	Absolute pressure:	140 bar
	Space velocity:	1.2 kg of slack wax per hour per litre of catalyst
	H <sub>2</sub> /oil ratio:	1750 standard litres of H <sub>2</sub> per kg of slack wax

After separation of the fraction boiling below 400°C and dewaxing of the fraction thus obtained, a lubricating oil was obtained with a viscosity index (VI<sub>E</sub>, ASTM-D 2270) of 139 and a kinematic viscosity at 99°C of 8.8 cSt.

The yield (based on the reactor feedstock) was 45% by weight.

#### WHAT WE CLAIM IS:—

1. A process for the preparation of lubricating oil with a high viscosity index, wherein a high-boiling mineral oil fraction is subjected to a catalytic hydrocracking treatment at elevated temperature and pressure and in the presence of a hydrocracking catalyst and hydrogen, the hydrocarbons boiling below a temperature between 350 and 400°C are separated from the liquid product of the hydrocracking treatment, the residue boiling above a temperature between 350 and 400°C is separated by dewaxing into lubricating oil with a high viscosity index and wax, said wax is subjected to catalytic hydro-isomerization in the presence of hydrogen and a catalyst containing one or more metals of the iron group, one or more metals of Group VIB of the Periodic Table of Elements, boron and a catalyst carrier consisting of oxide, and lubricating oil with a high viscosity index is separated from the catalytic hydro-isomerization product by dewaxing at least that part of said product which consists of hydrocarbons boiling within the boiling range of lubricating oils.

2. A process as claimed in Claim 1, wherein the high-boiling mineral oil fraction is a vacuum distillate boiling between 350°C and 550°C.

3. A process as claimed in Claim 1, wherein the high-boiling mineral oil fraction is a de-asphalted residual petroleum fraction or a

mixture thereof with a vacuum distillate boiling between 350°C and 550°C.

4. A process as claimed in any one of Claims 1—3, wherein the hydro-isomerization catalyst contains 3—16% by weight of one or more metals of the iron group, 6—24% by weight of one or more metals of Group VIB and 3—15% by weight of boron, calculated as B<sub>2</sub>O<sub>3</sub>.

5. A process as claimed in Claim 4, wherein the metal of the iron group is nickel and the metal of Group VIB is molybdenum.

6. A process as claimed in any one of Claims 1—5, wherein the hydrocracking catalyst contains 3—25% by weight of nickel and/or cobalt, 6—24% by weight of molybdenum and/or tungsten and 2—9% by weight of phosphorus, calculated as P<sub>2</sub>O<sub>5</sub>.

7. A process as claimed in Claim 6, wherein the only Group VB, VIB, VIIB or VIII metals present (as such and/or as oxides and/or sulphides) in the hydrocracking catalyst are nickel and tungsten.

8. A process as claimed in any one of Claims 1—5, wherein the hydrocracking catalyst is identical to the hydro-isomerization catalyst.

9. A process as claimed in any one of Claims 1—8, wherein the carrier of the hydrocracking and/or hydro-isomerization catalyst is alumina containing 0.5—3% by weight of silica and a maximum of 0.005% by weight of sodium.

10. A process as claimed in any one of Claims 1—9, wherein the hydro-isomerization catalyst contains fluorine, except when the hydrocracking catalyst is a catalyst as defined in Claim 6.

11. A process as claimed in Claim 10, wherein the hydrocracking and/or hydro-isomerization catalyst contains 1—6% by weight of fluorine.

12. A process as claimed in any one of Claims 1—11, wherein hydro-isomerization is carried out at a temperature between 310°C and 450°C, an absolute pressure between 90 and 200 bar, a space velocity between 0.1 and 5.0 litres of wax per hour per litre of catalyst and a hydrogen/wax ratio between 100 and 5000 standard litres per kg of wax.
13. A process as claimed in any one of Claims 1—12, wherein the hydrocracking process is carried out at a temperature between 350°C and 450°C, an absolute pressure between 100 and 200 bar, a space velocity between 0.1 and 5 kg of oil per hour per litre of catalyst and a hydrogen/oil ratio between 100 and 5000 standard litres of hydrogen per kg of oil.
14. A process as claimed in any one of Claims 1—13, wherein the fraction of the effluent from the hydro-isomerization zone boiling within the boiling range of lubricating oil is dewaxed and the wax thus obtained is recycled, at least in part, to the hydro-isomerization zone and/or the hydrocracking zone.
15. A process as claimed in any one of Claims 1—14, wherein the feedstock subjected to the catalytic hydro-isomerization process consists of a mixture of a high-boiling mineral oil fraction and wax obtained after the hydrocracking.
16. A process as claimed in Claim 8, wherein the hydrocracking and hydro-isomerization processes are combined in a single treatment carried out in a single reactor and using a single catalyst, by recycling the wax obtained after the dewaxing of the fraction of the effluent from the reactor boiling within the boiling range of lubricating oil.
17. A process for the preparation of lubricating oil with a high viscosity index as claimed in Claim 1 and substantially as hereinbefore described.
18. A lubricating oil with a high viscosity index whenever obtained by a process as claimed in any one of Claims 1—17.
- R. C. ROGERS,  
Chartered Patent Agent,  
Shell Centre,  
London, SE1 7NA.  
Agent for the Applicants.

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# ddonale

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CC228



Outlook

Estimated/Approved

Actual

Project Code: 8252

Project Name: MIDAS Replacement

Project Manager:

GIS Execution Owner: Merican, Fatimah

Project Status: Planned

[Click Here To Update Your Estimate History After Each Gate Change!](#)

Submit and Update Outlook

Undo all changes

Application  
ParentQuick Links: [2004]  
[2005]Summary  
Totals --

Overall Cost Report

Field Help

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you hit the SUBMIT button.

Add Years

Add Year

Baseline Changes

Benefits

(All Costs in KUSD)	Expense				Capital Expense					TO
	WF Months	WF Exp	NWF Exp	Total Exp*	Capital WF Months	Capital Staffing	Capital Software	Other CAPEX	Total CAPEX**	
Total:	0	700	0	700	0	0	0	0	0	700
IS Org:	0	700	0	700	0	0	0	0	0	700
IS Contract:	0	0	0	0	0	0	0	0	0	0
Infra Org:	0	0	0	0	0	0	0	0	0	0
Infra Contr:	0	0	0	0	0	0	0	0	0	0
Business:	0	0	0	0	0	0	0	0	0	0
Baseline Chgs Incl Above:	0	0	0	0	0	0	0	0	0	

Currency Exchange Rate Planning Basis (Forex rate used for your Gate 3 estimate)

Note: \*Total Expense = Workforce Expense + NonWorkforce Expense

\*\*Total CAPEX = Capital Staffing + Capital Software + Other CapEX

\*\*\*Total = Total Expense + Total CAPEX

Quick Links: [\[Summary\]](#) [\[2005\]](#)

-- 2004 --

[Overall Cost Report](#)[Field Help](#)

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## Expense

## Capital Expense

(All Costs in KUSD)	WF Months	WF Exp	NWF Exp	Total Exp*	Capital WF Months	Capital Staffing	Capital Software	Other CAPEX	Total CAPEX**	TOT
Total:	<input type="text" value="0"/>	<input type="text" value="350"/>	<input type="text" value="0"/>	<input type="text" value="350"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="350"/>
IS Org:	<input type="text" value="0"/>	<input type="text" value="350"/>	<input type="text" value="0"/>	<input type="text" value="350"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="350"/>
IS Contract:	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Infra Org:	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Infra Contr:	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Business:	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Comments:	<div></div>									

Last Updated: 5/12/03 12:45:00 PM

Submitter: Holtz, Alan

Yellow background cells represent calculated total.

Note: \*Total Expense = Workforce Expense + NonWorkforce Expense

\*\*Total CAPEX = Capital Staffing + Capital Software + Other CapEX

\*\*\*Total = Total Expense + Total CAPEX

Quick Links: [\[Summary\]](#) [\[2004\]](#)

-- 2005 --

[Overall Cost Report](#)[Field Help](#)

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[Calculate](#)[Delete](#)

## Expense

## Capital Expense

(All Costs in KUSD)	WF Months	WF Exp	NWF Exp	Total Exp*	Capital WF Months	Capital Staffing	Capital Software	Other CAPEX	Total CAPEX**	TOT
Total:	<input type="text" value="0"/>	<input type="text" value="350"/>	<input type="text" value="0"/>	<input type="text" value="350"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="350"/>
IS Org:	<input type="text" value="0"/>	<input type="text" value="350"/>	<input type="text" value="0"/>	<input type="text" value="350"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="350"/>
IS Contract:	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Infra Org:	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Infra Contr:	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Business:	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>	<input type="text" value="0"/>
Comments:	<div></div>									

**Last Updated:** 5/12/03 12:45:00 PM

**Submitter:** Holtz, Alan

Yellow background cells represent calculated total.

Note: \*Total Expense = Workforce Expense + NonWorkforce Expense

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